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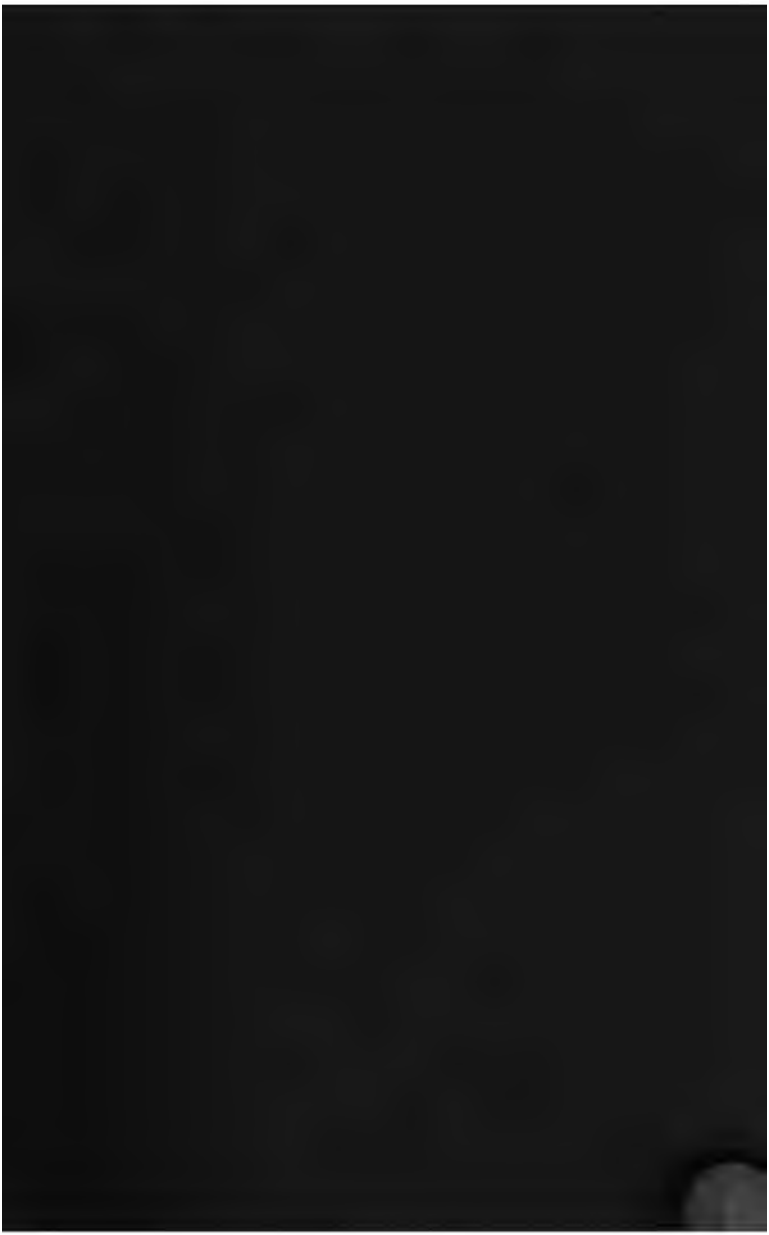
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PRACTICAL ORGANIC ANALYSIS

ELLIS.





PRACTICAL

ORGANIC ANALYSIS.

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AN INTRODUCTION TO

PRACTICAL

ORGANIC ANALYSIS:

ADAPTED TO THE REQUIREMENTS OF

THE FIRST M.B. EXAMINATION.

BY

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PREFACE.

THIS little book, though originally intended for the use of the medical student, will, I trust, prove equally serviceable as a general introduction to the study of Organic Analysis. The student who has passed his Preliminary Examination, and many others who have not yet reached that stage, are familiar with the ordinary processes of Inorganic Analysis, and therefore do not need a text-book which repeats a great part of that with which they are already acquainted.

In preparing this work, my task, of course, has been rather that of selecting from a large number of well-known reactions than that of furnishing new ones. The general arrangement of the work, however, together with much that it contains, will, I believe, be found to be new.

It has not been found advisable to burden the student with too large a number of tests for each individual substance, and those reactions have been introduced which will prove of use in general ana-

lytical work, to the exclusion of many which are merely interesting.

I have also found the exercises afforded by the examination of Organic Matter supposed to contain simple poisons both useful and instructive.

For much that is contained in this little book I am indebted to the teaching of Professor A. W. Williamson, of University College, London, to whom I beg to tender my grateful acknowledgments. Confirmatory assistance has also been derived from the valuable stores of information contained in Watts' 'Dictionary of Chemistry,' and in Fresenius' 'Qualitative Analysis.'

I shall esteem it a favour if teachers and students will forward me notes of difficulties and emendations, in order that the work may become as complete and as trustworthy as possible.

G. E. R. E.

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PRACTICAL ORGANIC ANALYSIS.

SECTION I.

PRELIMINARY EXAMINATION OF A SUBSTANCE FOR AN OXALATE, ACETATE, BENZOATE, SUCCINATE, TARTRATE, CITRATE, MALATE, MECONATE, HIPPURATE, URATE, GALLATE, TANNATE, CYANIDE OR DOUBLE CYANIDE, OR FOR ALCOHOL, ETHER, STARCH, CANE SUGAR, GRAPE SUGAR, GLYCERINE, CHLOROFORM, UREA, MORPHIA, STRYCHNIA, QUINIA, OR CINCHONIA.

MUCH information can generally be obtained by a careful preliminary examination.

(A) **The substance is a Liquid.**

1. It may be a solution of an **organic salt,** or **acetic** or **hydrocyanic acid,** or **ether, alcohol, glycerine,** or **chloroform.**

A. Carefully evaporate a portion of the liquid in a porcelain dish, note the appearances presented on drying; do not bake the residue (if any), as this might cause decomposition.

(*a*) A residue is left. Probable presence of **an organic salt**; see **7.**

(β) No residue is obtained. Probable presence of **acetic** or **hydrocyanic acid,** or of **alcohol, ether, glycerine,** or **chloroform.**

2. To a portion of the liquid add dilute H_2SO_4 and warm. A smell of vinegar indicates the presence of an **acetate**; a faint smell of peaches indicates the presence of **hydrocyanic acid.**

3. *B.* Note the odour of the liquid. **Ether** and **alcohol** may be thus detected, unless much diluted with H_2O, as is sometimes the case. If the presence of either be suspected proceed to **41.**

4. *C.* Taste the liquid. If it be sweet, and if it left no residue, either **chloroform** or **glycerine** is present. If the liquid burns with a luminous flame on heating, glycerine is probably present. Confirm by **44.** Chloroform boils at 62°; ether at 35°; alcohol at 78·5°; glycerine at a temperature much above 100°.

D. If no result has been obtained, and the liquid is neutral, probably H_2O alone is present.

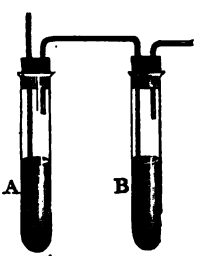
5. The presence of organic matter in any case may be shown thus:—Test the liquid with litmus-paper; if it be acid, add NaHO (or KHO); if alkaline, add acid (HCl) till the liquid is neutral. [See that the alkali does not contain CO_2; if so, add the NaHO in quantity not quite sufficient to neutralize the liquid; on evaporating, the residue will be free from CO_2.] Evaporate down. Then, using the apparatus in fig. 1, in A put some strong H_2SO_4 and $K_2Cr_2O_7$ (powdered) so as to form a thin mud, then

add the residue obtained above. In B put some lime water. On heating the mixture in A, CO_2 will be evolved if the residue contains **carbon,** and will be detected by the lime water becoming milky.

$$H_2SO_4 + K_2Cr_2O_7 = K_2SO_4 + H_2Cr_2O_7.$$

The chromic acid, thus formed, gives off O (nascent) on being heated, and this nascent oxygen combines vigorously with any C present, forming CO_2.

FIG. 1.



6. The presence of organic nitrogen (*i.e.* not in the form of ammonia) in a body is detected thus :—If the substance be an ammonium salt, remove the H_3N by boiling it with an excess of KHO, evaporate and mix the residue with soda lime; introduce the mixture into the hard tube A (fig. 2), put a little more soda lime on the top of the mixture, also a small quantity of H_2O in C, and heat A. If **organic nitrogen** be present in A, ammonia will be given off, and will be absorbed by the H_2O in C. Its presence may be detected by Nessler's test.

(B) **The substance is a Solid.**

7. *A.* Heat a portion of the solid in a hard glass tube, or porcelain crucible. Note if there is any charring, and if any smell is perceptible.

(*a*) MUCH CHARRING. Presence of **tartrate,**

citrate, gallate, urate, tannate, cane or **grape sugar, starch,** or an **alkaloid.**

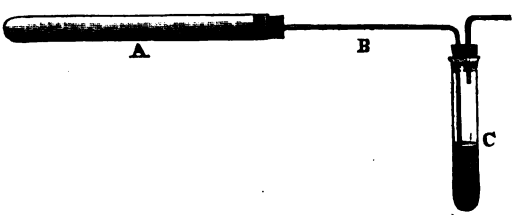
1. If also there is a smell of burnt feathers and of ammonia, a **urate** is probably present.

2. If a tarry smell be noticed, an **alkaloid** is probably present.

3. The smell of burnt sugar indicates the presence of a **tartrate, citrate, gallate, tannate, cane sugar, grape sugar,** or **starch.**

If either starch or an alkaloid be suspected, pre-

FIG. 2.



pare a solution of the solid as directed in **9.** To a portion of the solution add a little of a solution of iodine in potassium iodide.

A dark-blue colour (almost black) shows the presence of **starch** ; see **52.**

[This is due to the formation of the so-called iodide of starch.]

A brown precipitate shows the presence of an **alkaloid.**

(β) LITTLE OR NO CHARRING. Presence of an

acetate, benzoate, succinate, malate, oxalate, sulpho- or **double cyanide,** or **urea.**

1. A smell of burnt sugar and of ether indicates the presence of an **acetate.**

2. A smell of burnt feathers and of ammonia indicates the presence of a **cyanide** or **double cyanide.**

3. A smell of ammonium sulphide shows that a **sulpho-cyanide** is present.

4. Ammonia is given off, and a white sublimate is formed on the sides of the tube. **Urea** is present.

5. The solid volatilises, but there is no smell. Probable presence of an **oxalate.**

6. A peculiar aromatic odour is perceived. A **benzoate** is present.

7. A gas with a stifling odour is evolved. Probable presence of a **succinate.**

8. The same with a strong smell of burnt sugar indicates the presence of a **malate.**

8. B. Heat the dry solid in a test-tube with strong H_2SO_4.

(α) The mixture does not blacken. **Tartrates** and **citrates** are absent.

(β) A gas is evolved which burns with a blue flame (CO) at the mouth of the tube. Presence of **malate, formate, oxalate, ferro-cyanide,** or **ferri-cyanide.**

1. If the original solution was greenish and becomes white after the addition of the acid, probably a **ferro-** or **ferri-cyanide** is present.

2. If a smell of fruit is perceived, a **malate** is probably present.

(γ) A smell of vinegar is noticed. An **acetate** is present.

(δ) SO_2 is evolved, as also an irritating, cough-causing vapour. A **succinate** is present.

(ϵ) If in addition CO_2 is given off, but no charring takes place, a **urate** is probably present.

(ζ) Much CO_2 and SO_2 are evolved, and the mixture chars rapidly. A **tartrate, sugar,** or **starch** is present.

(η) No charring, but an irritating, cough-causing vapour is evolved. A **benzoate** is probably present.

(θ) Charring takes place, CO_2 and CO are evolved. A **citrate** is probably present.

(ι) The mixture darkens rapidly, and an irritating smell is noticed. A **gallate** or **tannate** is present.

If the liquid possesses a greenish tint, then a **tannate** is present; if a purplish tint, then a **gallate** is present.

SECTION II.

SPECIAL TESTS FOR THE ACIDS AND OTHER ORGANIC BODIES.

FOR these tests it is best to separate from the solution any metal which it may contain (except K, Na, or $\overline{Am}$), as the reagents used are liable to precipitate some of the metals from their solutions.

9. This is generally done by boiling the given solution with Na_2CO_3 (solution) until no further precipitate is obtained. Filter off the precipitate,[1] and to the filtrate add a very slight excess of HNO_3, boil to expel any CO_2 present, then add NaHO, drop by drop, till the solution is exactly neutral. The solution thus obtained is then used for the following tests, and is referred to as the original 'Na_2CO_3' solution.

10. Formic Acid and Formates: CH_2O_2.
Acetic Acid and Acetates: $C_2H_4O_2$.

These two acids, corresponding respectively to methyl and ethyl alcohol, belong to the fatty acid series. Formic acid, found in nettles and in the bodies of red ants, is a fuming irritating liquid, boiling

[1] This treatment with Na_2CO_3 may be omitted when metals of the first four groups are absent.

at 105°. Acetic acid, obtained from vinegar, is a colourless liquid, with a strong acid taste and smell, boiling at 117°, and freezing at 17°. The acetates of silver and mercury (protosalt) are only slightly soluble in water; the other acetates are readily soluble.

11. *Tests.*—Formic Acid.

1. Heated with strong H_2SO_4, no charring takes place, but CO is evolved, which, if lighted, burns with a blue flame. [An oxalate reacts in the same way.]

2. To a neutral solution of a formate, add C_2H_6O, then strong H_2SO_4; heat. Formic ether $\left(\begin{matrix}CH_3\\CH_3\end{matrix}\right\} O\Big)$ is given off (fragrant smell). If an acetate be used, acetic ether will be produced, but the smells are so alike that this test cannot be relied upon to distinguish between the two salts.

3. $AgNO_3$ gives no precipitate with a formate except when the solution is concentrated. Now heat; the formate of silver is reduced, and grey Ag is deposited. An acetate does not thus reduce $AgNO_3$.

4. Fe_2Cl_6, added to a neutral solution of either a formate, acetate, or sulpho-cyanide, gives a red colouration. Divide the solution into two portions, (α) and (β). Heat (α); a precipitate is produced in the case of the formate and acetate. To (β) add HCl. If the colour changes to yellow, a formate or acetate is present. Distinguish thus: A formate gives a white

precipitate with mercuric chloride; an acetate gives no precipitate.

5. $BaCl_2$ (or $CaCl_2$) gives no precipitate with a formate solution.

6. Add HgO to some formic acid; mercuric formate is thus produced, and CO_2 is given off. Now heat the solution; the formate is reduced, and a grey powder (Hg) is deposited. Mercuric acetate is not thus reduced.

Both formic and acetic acid are monobasic, as determined by the following—

12. *Rule for determining the Basicity of Organic Acids.* It is found that the basicity of an organic acid is determined by the number of times it contains the group COOH (Oxatyl). Thus formic acid, H (COOH), is monobasic, containing it once. Succinic acid is dibasic, $C_2H_4\begin{cases}COOH\\COOH,\end{cases}$ while citric acid contains it three times, C_3H_4 (OH) $(COOH)_3$, and is therefore tribasic.

13. *Tests.*—Acetic Acid.

1. On heating an acetate with strong H_2SO_4, no charring takes place until the heating has been continued some time. Much charring then occurs, and $H\bar{A}$ is set free, and may be recognised by its smell (of vinegar). Acetic ether, which possesses a fragrant smell, is given off when C_2H_6O and strong H_2SO_4 are added to an acetate solution, and the mixture warmed (see **11**, 2). The odour is best observed

when the mixture is allowed to cool for a short time.

2. To the neutral solution of an acetate add $AgNO_3$. A white crystalline precipitate of silver acetate ($C_2H_3AgO_2$) is produced, soluble in (NH_4) HO, and in warm water.

3. Fe_2Cl_6, added to a neutral acetate solution, produces a deep red colouration (but no precipitate), due to formation of iron peracetate. [The colour is not destroyed by $HgCl_2$, which destroys the red colour due to the presence of a sulpho-cyanide; see **39**, 3.] Now boil; all the Fe separates as a light brown precipitate (basic acetate of Fe), and the liquid becomes colourless.

4. A little acetate (solid), heated in a hard glass tube, gives off the peculiar smell of acetone. The vapour burns with a luminous flame when kindled.

5. $BaCl_2$ (or $CaCl_2$) gives no precipitate with a neutral solution of an acetate.

14. Benzoic Acid and Benzoates: C_6H_5. COOH.
Succinic Acid and Succinates:

C_2H_4. $(COOH)_2$.

Benzoic acid is monobasic, and consequently forms only one series of salts, most of which are soluble in water. Succinic acid, belonging to the oxalic series of acids (general formula $=C_nH_{2n-2}O_4$), is dibasic, and thus forms two series of salts, of which the alkaline metal salts are soluble in H_2O. Benzoic

acid is much less soluble in water than is succinic acid.

15. *Tests.*—Benzoic Acid.

1. Heated alone a benzoate does not char, but gives off a vapour possessing an aromatic odour, which causes an unpleasant sensation and induces coughing. Benzoic acid crystals burn with a smoky flame, while succinic acid crystals burn with a blue but not a smoky flame.

2. When heated with concentrated H_2SO_4, no charring occurs, but an irritating, cough-causing vapour is evolved.

3. $AgNO_3$ gives a white precipitate of silver benzoate.

4. Fe_2Cl_6, added to a neutral solution, gives a buff-coloured precipitate (ferric benzoate). This precipitate is soluble in acids with liberation of benzoic acid (white, crystalline flakes), and is decomposed by NH_4HO.

5. When concentrated H_2SO_4 (or HCl) is added to a fairly strong benzoate solution, benzoic acid is liberated in the form of a white crystalline precipitate. [Benzoic acid is slightly soluble in H_2O.]

16. *Tests.*—Succinic Acid.

1. Heated alone, little or no charring takes place, but cough-causing vapours are evolved. (Compare **15**, 1).

2. When heated with concentrated H_2SO_4, much SO_2 is given off, but no blackening takes place until

the heat has been long prolonged, when an irritating, cough-causing vapour is evolved.

3. $AgNO_3$ gives a white precipitate of silver succinate.

4. Fe_2Cl_6 gives a reddish-brown precipitate (ferric succinate) with neutral succinates. Ferric succinate is soluble in HCl (without liberation of succinic acid), and is decomposed by NH_4HO with formation of ammonium succinate and a more basic succinate. [Fe_2Cl_6 is an acid liquid, so care must always be taken to add it drop by drop.]

5. $CaCl_2$ gives no precipitate with either a benzoate or a succinate.

6. To the neutral succinate solution add an equal volume of C_2H_6O, then a drop of NH_4HO, and $BaCl_2$. A white precipitate of barium succinate is formed, this salt being insoluble in C_2H_6O. As barium benzoate is soluble in alcohol, this test serves to distinguish between a benzoate and a succinate.

17. Oxalic Acid and Oxalates: $(COOH)_2$.
Tartaric Acid and Tartrates: $C_4H_6O_6$.
Malic Acid and Malates: $C_4H_6O_5$.
Citric Acid and Citrates: $C_6H_8O_7$.

Oxalic acid is the first term of a series of dibasic acids possessing the general formula $C_nH_{2n-2}O_4$. The oxalates of the alkaline metals are soluble in H_2O, the other metallic oxalates are generally insoluble (stannic oxalate is, however, soluble). Malic and tar-

taric acids are likewise dibasic, and consequently form two series of salts; thus, tartaric acid, $\left.\begin{matrix}H\\H\end{matrix}\right\} C_4H_4O_6$, forms two salts with K; the one, hydrogen potassium tartrate (cream of tartar), $\left.\begin{matrix}K\\H\end{matrix}\right\} C_4H_4O_6$; the other, potassium tartrate, $\left.\begin{matrix}K\\K\end{matrix}\right\} C_4H_4O_6$. The malates generally are soluble in H_2O. Citric acid occurs together with malic acid in the juice of many fruits (such as the lemon). The citrates of the alkaline metals are soluble in H_2O, while those of the alkaline-earth metals (Ca, Ba, Sr), of silver and of lead are insoluble.

The acids, tartaric, malic, and citric, possess the property, in common with other complex organic bodies, of holding metallic oxides (as CuO) in solution, even in the presence of an alkali. This is due to the formation of a double tartrate (malate or citrate) of the alkali and the metal of the oxide, which salt is not decomposed by the alkali.

The formula of tartaric differs from that of succinic acid by O_2, and we can obtain the latter acid from the former, and *vice versâ*. Of the six atoms of O in the formula of tartaric acid, four correspond to the bibasic function of the acid (see **12**); the other two atoms of O are united in what is termed an alcoholic bond, and these two O's hold the CuO in solution.

18. *Tests.*—Oxalic acid.

1. On heating oxalic acid in a hard tube the acid partly sublimes, condensing in fine needles, and is

partly decomposed. Vapours are given off which induce coughing.

2. When a solid oxalate is heated with concentrated H_2SO_4 no charring takes place, but an effervescence is caused, due to the evolution of CO and CO_2. The CO burns with a blue flame when a light is applied, and the CO_2 renders milky a drop of lime water on the end of a glass rod. An oxalate is not decomposed by dilute H_2SO_4.

3. Heat a strong solution with MnO_2 and dilute H_2SO_4 ; CO_2 gas is given off.

N.B.—See that the MnO_2 does not itself contain CO_2 (a carbonate, which the commercial sample generally does), which would, of course, give off CO_2 on being treated with an acid.

4. On adding $CaCl_2$ to a solution of an oxalate, a white precipitate of calcium oxalate (CaC_2O_4) is produced, insoluble in NH_4HO, and in acetic acid, but easily soluble in HCl or HNO_3. [This precipitate is obtained even if the solution be very dilute.]

Filter off the CaC_2O_4 thus obtained ; divide it into two parts, (*a*) and (*b*). To (*a*) add dilute HCl ; no effervescence occurs. Dry (*b*) and heat it to redness on Pt foil ; no blackening occurs, but the CaC_2O_4 is converted into $CaCO_3$, which, of course, effervesces on adding dilute HCl.

5. $CaSO_4$ (or lime water, or $BaCl_2$) added to an oxalate reacts as in 4.

6. If a few drops of Fe_2Cl_6 be added to a

neutral oxalate solution, no precipitate is formed, but on now adding AmHO (or KHO) till the liquid is strongly alkaline, a dark brown precipitate is thrown down. (Compare **19**, 7.)

7. $AgNO_3$ added to an oxalate solution produces a white precipitate of $Ag_2C_2O_4$, soluble in AmHO and in HNO_3.

19. *Tests.*—Tartaric Acid.

1. Heated alone, tartrates are decomposed, a characteristic smell of burnt sugar is given off, and much charring occurs.

2. When heated with strong H_2SO_4, these salts blacken rapidly, giving off CO, CO_2, and SO_2. [The amount of CO given off is small.]

3. Add $CaCl_2$ to a neutral tartrate solution, shake well, and let stand; a dense white precipitate of calcic tartrate (compare citrates and malates, **20**, **21**, 3) is formed, soluble in most acids (so that no precipitate is got with tartaric acid until the acid has been neutralized), and in salts of $\overline{Am}$. Heat, to coagulate the precipitate, wash and divide into two parts (*a*) and (*b*). To (*a*) add cold KHO, the precipitate is dissolved; now heat, the calcium tartrate is reprecipitated, but is redissolved on cooling.

Pour off as much wash-water as possible from (*b*), add dilute AmHO, drop by drop, until the $Ca\overline{T}$ is nearly dissolved, drop in a crystal or two of $AgNO_3$, then heat gently to about 55°–60° C. (best in a water bath). A bright mirror of metallic silver is seen inside

the tube. $H_2\overline{T}$ reduces Ag while still in the wet tube, and thus differs from other bodies which also reduce Ag salts.

4. CaHO in neutral solutions causes a white precipitate of $CaC_4H_4O_6$, soluble in NH_4Cl and in $H_2\overline{T}$. This precipitate, of course, reacts in the same way as (*a*) and (*b*) in 4.

5. $AgNO_3$ produces in neutral solutions a curdy white precipitate of Ag_2T, soluble in HNO_3 and AmHO. Wash the precipitate, drain off the wash-water, and add dilute AmHO till the precipitate is nearly dissolved, heat gradually (best in a water-bath) to 55°–60° C. ; a brilliant deposit of Ag is seen on the inside of the tube (as in 4 *b*).

6. Add ($KCl + H\bar{A}$) to a neutral solution of a tartrate, shake and stir well. A white crystalline precipitate of $KH\overline{T}$ is formed, soluble in mineral acids (HCl, HNO_3, &c.) and KHO, but not in acetic acid. The formation of the precipitate is aided by the addition of C_2H_6O. The $H\bar{A}$ was added to make the solution acid, as the precipitate is not produced in neutral solutions or in solutions containing a mineral acid.

7. As before stated, tartaric acid (and other organic bodies) are capable of holding Cu and other metals (Al, Mn, Fe) in solution in the presence of alkalis. Of course, there is a limit to the quantity of the CuO (or other oxide), which these bodies can prevent from being precipitated, so always use a little. If an excess has been added, of course part of the

oxide will be precipitated, but some will dissolve in the acid, and its presence be proved (in the case of CuO) by looking through the edge of the solution and seeing if the clear part of the solution has a bluish tint. Proceed thus—

To some KHO solution add a little $CuSO_4$, a light blue precipitate of $Cu(OH)_2$ is formed; now add $H_2\overline{T}$ or a neutral tartrate solution, the blue precipitate is dissolved, and a blue solution is obtained.

The acidity of $H_2\bar{T}$ is less pleasant than that of citric acid.

20. *Tests.*—Malic Acid.

1. Heated to redness alone, malates darken very slowly, giving off a smell of burnt sugar as well as an irritating vapour.

2. Heated with strong H_2SO_4, the mixture darkens very slowly, giving off CO and CO_2, a smell of apples, and afterwards SO_2

3. $CaCl_2$, when added to a neutral malate solution, gives no precipitate in the cold, nor upon boiling (unless the original solution be very strong). Now add an equal volume of C_2H_6O. A white precipitate of $C_4H_4CaO_5$ is thrown down. This calcium malate, however, gives no mirror when heated with a crystal of $AgNO_3$ and a little AmHO (see **19**, 3).

4. Lime water gives no precipitate with malates either in the cold or on boiling. Now add C_2H_6O, and we get a precipitate of calcium malate, as in 3. Hence we can distinguish between a tartrate and a malate.

5. To a neutral solution of a malate add PbA_2. A white precipitate of lead malate is thrown down. Now boil the liquid (containing the precipitate); the lead malate seems to dissolve, while the particles seem to clot together and undergo a kind of fusion.

This is not a characteristic test, but no other common acid reacts in the same way.

6. $AgNO_3$, added to neutral solutions, throws down a granular white precipitate of $Ag_2C_4H_4O_5$, which turns grey on boiling.

21. *Tests.*—Citric Acid.

1. On being heated to redness citrates char considerably, and give off irritating fumes, which are readily distinguished from those given off by tartrates.

2. When heated with strong H_2SO_4, CO and CO_2 are given off, but no blackening takes place until after long boiling, when SO_2 also is evolved.

3. $CaCl_2$ gives no precipitate with a neutral solution of a citrate in the cold, but on boiling the solution, a white precipitate of calcium citrate, $Ca_3(C_6H_5O_7)_2$, is thrown down, soluble in AmCl or AmHO, but insoluble in KHO (except when strong and in the cold).

4. Lime water gives a precipitate (as in 3), when boiled with a neutral solution of a citrate. Citrate of lime does not reduce a silver salt as calcium tartrate does.

5. $AgNO_3$, added to a neutral solution, produces a white precipitate of silver citrate, $C_6H_5Ag_3O_7$.

Silver citrate dissolves in AmHO, but does not darken on boiling (owing to the reduction of the silver) as silver tartrate does.

22. Meconic Acid and Meconates: $C_7H_4O_7$.

Meconic acid, found in opium, is a tribasic acid, soluble in warm H_2O, from which it may be crystallized in the form of plates. The normal meconates (those in which the three atoms of H are replaced by a metal) of the alkaline metals are soluble in water. The acid meconates are only slightly soluble, while silver, lead, barium, calcium, and copper meconates are insoluble in H_2O.

23. *Tests.*

1. Meconates are readily decomposed on boiling, CO_2 being evolved.

2. $CaCl_2$ forms a precipitate of calcium meconate with a neutral solution. The precipitate is soluble in $H\bar{A}$.

3. Fe_2Cl_6 gives a blood-red colouration (resembling that due to the action of Fe_2Cl_6 on a sulphocyanide). The solution is not discolourised on adding $HgCl_2$, as is the case with a sulphocyanide, and does not alter in appearance on boiling, as would be the case with an acetate solution.

This reaction is highly characteristic of a meconate.

4. With $Pb\bar{A}_2$ meconates give a white precipitate of lead meconate, which is insoluble in $H\bar{A}$.

24. Uric Acid and Urates: $C_5H_4N_4O_3$.
Hippuric Acid and Hippurates: $C_9H_9NO_3$.

These two acids differ from the ones we have already dealt with by containing N. Uric acid is obtained from the excrement of birds and serpents, and is a common constituent of animal excretions, either as uric acid or urate of sodium, potassium, ammonium, or calcium. Serpent's urine (hydric ammonium urate) is nearly insoluble in H_2O, but more soluble in KHO (dilute). From this solution hydric urate is precipitated by the addition of HCl. Uric acid is insoluble in H_2O, ether, and C_2H_6O, but soluble in dilute alkalis and in concentrated H_2SO_4.

Hippuric acid is found in the urine of horses and other herbivorous animals. It very closely resembles benzoic acid in its reactions. In its passage through the animal body benzoic acid becomes converted into hippuric acid.

All the urates are only slightly soluble in H_2O.

25. *Tests.*—Uric Acid.

1. When heated alone, urates blacken readily, and give off a smell of burnt feathers; sometimes the smell of HCN may also be detected.

2. Heated with strong H_2SO_4, the solution does not blacken, but at a high temperature SO_2 and CO_2 are given off.

3. Add HCl, drop by drop, to the neutral solution. A white crystalline precipitate forms gradually. Take the precipitate, dissolve it in a few drops of HNO_3,

evaporate carefully to dryness, and add a little H_4NHO (one drop). A beautiful purple-red colour is formed, due to the formation of murexide, $C_8H_4(NH_4)N_5O_6$.

Acetic acid also precipitates uric acid from its solutions.

4. Uric acid readily dissolves in HNO_3, and the bright purple colour (murexide ; see 3) is obtained by evaporating the solution, and moistening the residue with AmHO. By acting on uric acid with HNO_3, alloxantin ($C_8H_4N_4O_7$) is formed, which on drying gives alloxan ($C_4H_2N_2O_4$), and this substance gives the purple colour with AmHO, which colour becomes violet on the further addition of KHO.

5. $AgNO_3$ gives a black precipitate with a urate solution. Schiff's test consists in dissolving the hydric urate (precipitated by HCl, as in 3) in Na_2CO_3. A drop of this solution is placed alongside a similar drop of $AgNO_3$ on blotting paper. On now warming, a brown stain is caused, due to the reduction of the silver salt by the uric acid.

26. *Tests.*—Hippuric Acid.

1. Heated alone, the acid chars, a sublimate of benzoic acid is obtained, and vapours with a characteristic odour are evolved. The smell of HCN is generally perceptible.

2. The hippurates very closely resemble the benzoates in their reactions. The only methods of distinguishing between them are :—

(*a*) HCl, in strong solutions of hippurates and benzoates, gives a white precipitate (if it does not form at once, shake the solution well). On adding a little ether, the benzoate precipitate dissolves, while the hippurate one does not. The acids when mixed may be separated in this way, the ethereal solution rising to the surface.

If a hippurate be boiled with HCl, benzoic acid and sugar of blue are formed, thus :—

$$H_2O + C_9NH_9O_3 = C_7H_6O_2 + C_2H_5O_2N$$
$$= C_6H_5COHO + \begin{cases} CH_2HO \\ CONH_2 \end{cases}$$

(*b*) On heating a hippurate with soda lime, we get H_3N given off, which is not the case with a benzoate (see **6**).

3. Fe_2Cl_6 gives a brown precipitate with neutral solutions of hippurates, as it does with benzoates.

27. Bile Salts.

Purified bile is a mixture of the sodium salts of taurocholic ($C_{26}H_{45}NSO_7$) and cholic acids ($C_{26}H_{43}NO_6$). These salts resemble soaps, for, on boiling with Glauber's salts (Na_2SO_4), the precipitated bile salts rise to the surface along with the colouring matter. Strong H_2SO_4 forms taurin ($C_2H_7NO_3S$) with taurocholic acid, and with cholic acid it gives cholanic acid ($C_{24}H_{40}O_5$), which gives a fluorescing liquid with sugar and oil of vitriol. On addition of an acid the bile salts give the hydric salts. The soda salts are soluble in C_2H_6O ; on now

adding ether, the salts are deposited with most of the colouring matter; a second addition of ether will bring down the salts free from colouring matter.

28. *Tests.*—Bile Salts.

On adding H_2SO_4, drop by drop, to a tolerably dilute aqueous solution of bile salts, a white precipitate is formed, which dissolves on an excess of the acid being added. Now pour in a very little cane sugar. A beautiful violet colouration is produced, due to the cholanic acid liberated in the reaction. (Pettenkofer's test.)

Too much sugar causes a black colouration.

29. Tannin or Tannic Acid } **and Tannates**: $C_{27}H_{22}O_{17}$.

Gallic Acid and Gallates: $C_7H_6O_5$.

Both these bodies are obtained from gall-nuts (excrescences formed on the oak tree by an insect). Powdered gall-nuts, on being treated with ether and H_2O, yield the tannin to the H_2O, and the gallic acid to the ether. Tannin is not a real acid, because it contains no oxatile, but is in reality a salt of gallic acid (gallic gallate). When exposed to the air, or treated with dilute H_2SO_4, tannin breaks up into glucose ($C_6H_{12}O_6$) and gallic acid, thus :—

$$C_{27}H_{22}O_{17} + 4H_2O = C_6H_{12}O_6 + 3C_7H_6O_5,$$

and when heated to about 220°, it yields pyrogallic acid, $C_6H_3(OH)_3$. In pyrogallic acid, each of the three atoms of O is interposed between C and H, and thus

we have three atoms of hydroxyl. On being heated, gallic acid is decomposed into CO_2 and $C_6H_3(OH)_3$.

30. *Tests.*—Tannic Acid.

1. Heated alone, tannates char easily, and give off a strong smell, resembling burnt sugar.

2. Heated with strong H_2SO_4, a dark mixture with a greenish tint is obtained : vapours are evolved which induce coughing. A similar reaction is obtained with a gallate (see **31**, 2).

Heated with dilute H_2SO_4, grape sugar and gallic acid are formed.

3. $CaCl_2$ gives no precipitate with a tannate solution.

4. H_2SO_4 gives a white curdy precipitate.

5. Fe_2Cl_6 added to a solution of a tannate gives a dark blue-black precipitate (ink). The precipitate (tannate of iron) dissolves in H_2SO_4, and the solution then becomes yellow.

6. Gelatin gives a yellowish-white flocculent precipitate (of leather).

7. When KHO is added to a tannate solution (neutral), the mixture gradually becomes yellowish-brown, and then black. The reaction proceeds more slowly than in the case of a gallate solution.

31. *Tests.*—Gallic Acid.

1. When heated alone, gallates char easily. A faint smell of burnt sugar may sometimes be noticed.

2. Heated with strong H_2SO_4, a dark mixture is obtained, which gives off unpleasant vapours, and in which a dark crimson tint may be noticed.

3. $CaCl_2$ gives no precipitate.

4. H_2SO_4 gives no precipitate.

5. Gelatin also gives no precipitate.

6. Fe_2Cl_6 gives a blue-black precipitate (ink), which forms a dark yellow solution on adding H_2SO_4.

7. A mixture of KHO and gallic acid slowly turns yellowish brown, and finally black. This blackening with gallic acid takes place more rapidly than with tannin, and less quickly than with pyrogallic acid. A kind of crimson tint may be observed in the gallic acid colouration.

32. The separation of tannin and gallic acid, occurring together in solution, is thus effected :— Evaporate the solution gently, until it is very concentrated. Let cool, and then shake it up in a narrow test-tube with ether. We now get two layers, the upper one of ether and gallic acid, the lower of tannin and water. Introduce a small roll of blotting-paper and remove, by means of it, the ethereal solution. The gallic acid is thus got on the paper, and the tannin is left in the test tube.

33. Urea : CON_2H_4.

Urea is a white crystalline solid, occurring in the form of long striated needles, readily soluble in H_2O and C_2H_6O. It occurs in varying quantity in the urine of mammals, and may also be prepared artificially. Heated in closed tubes with H_2O, urea forms AmHO and CO_2. When subjected to dry

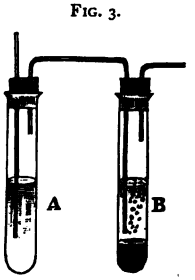
distillation, it behaves as a cyanurate of ammonia, giving off NH_3, while cyanuric acid ($C_3N_3O_3H_3$) is left behind. Cyanuric acid, when heated, breaks up into three molecules of cyanic acid (CNOH). Although neutral, urea, like many amides, is capable of combining feebly with acids and bases; thus we have urea nitrate, $\left.\begin{matrix}CONH\\NH_3\end{matrix}\right\}HNO_3$. When exposed to the air, urea very soon becomes converted into ammonium carbonate.

34. *Tests.*—Urea.

1. Heated alone in a hard tube, the solid melts. Ammonia is given off (recognised by its smell and action on litmus), and a white sublimate of cyanuric acid is formed on the sides of the tube.

2. When HNO_3 is added to a strong solution of urea, urea nitrate is formed, which, being almost insoluble in HNO_3, separates out in shining scaly plates. Urea nitrate is soluble in C_2H_6O and hot H_2O.

FIG. 3.



3. Put some of the precipitate, obtained in 2, in the test-tube A (fig. 3), and add some HNO_2; the precipitate dissolves, CO_2 and N are given off. The CO_2 gives a white precipitate with the lime water in B.

$$CON_2H_4 + 2HNO_2 = CO_2 + N_4 + 3H_2O.$$

4. $Hg(NO_3)_2$ gives a whitish-grey curdy precipitate with urea.

5. Add KHO to urea solution, and heat. The urea is decomposed, and NH_3 is evolved.

35. Hydrocyanic Acid and Cyanides: HCN.
Ferrocyanic Acid and Ferrocyanides: H_4FeCy_6.
Ferricyanic Acid and Ferricyanides: H_3FeCy_6.
Sulphocyanic Acid and Sulphocyanides: HCyS.

The most common salt of these acids is the double cyanide of potassium and iron (yellow prussiate of potash, $K_4FeC_6N_6$), from which, directly or indirectly, all the other cyanides are prepared. The simple cyanides (as KCN, HCN) are very unstable, while the double cyanides of Fe, Cr, Co, and other metals are very stable. Hydrocyanic acid cannot be kept in a pure state (free from H_2O), as it is gradually converted into ammonium formate. This action takes place at once if the acid is heated to boiling with KHO.

$$HCN + 2H_2O = NH_3 + C\acute{O}_2H_2.$$

HCN possesses a peculiar characteristic smell of bitter almonds. The cyanides of the alkaline and alkaline earth metals, as also of gold, mercury, and iron, are soluble in H_2O. Cadmium cyanide also is slightly soluble, while the cyanides of the other metals are insoluble.

In the ferrocyanides and ferricyanides (as also in the case of the double cyanides of Co) the Fe is

present as a chlorous or non-metallic radicle, and therefore does not give the ordinary reactions for iron; consequently $FeCy_6$ is the chlorous part of the salt, and K the metallic part.

The sulphocyanides correspond to the cyanates; the O of the cyanates being replaced by S in the sulphocyanides.

36. *Tests.*—Hydrocyanic Acid.

1. Heated alone, cyanides (or double cyanides) do not blacken (or very slightly), but traces of AmHO may be detected.

2. When heated with dilute H_2SO_4, hydrocyanic acid is set free, and may be recognised by its smell of bitter almonds. [As HCN is *very* poisonous, this test should be performed very carefully, and with a very little of the solid cyanide. Care should be taken not to inhale the vapour.]

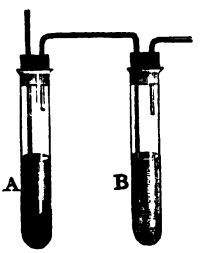
3. To a solution of a cyanide, add a few drops of KHO, and then a little $FeSO_4$ containing a little ferric salt; a brownish-green precipitate is obtained (Prussian blue and ferrous and ferric hydrate). Now boil, and acidify the solution with HCl; the brown colour, due to the two hydrates of iron, disappears, but a bluish-green precipitate of $K_2Fe_4Cy_{12}$ (Prussian blue) remains. If the cyanide were present in small quantity, the Prussian blue would dissolve, after the addition of HCl, and a bluish-green solution would be obtained.

4. $AgNO_3$ gives a white precipitate of AgCy,

soluble in much AmHO, and in excess of KCy, insoluble in HNO_3. Heat the AgCy precipitate; it decomposes, and metallic silver remains. The precipitate which $AgNO_3$ gives with a chloride is not thus decomposed.

5. Introduce into the test-tube A (fig. 4) the cyanide solution, and then some dilute HCl. In B put some Am_2S solution. Now, on heating A, HCN is given off, and passes into the solution in B, forming AmCyS. Boil the solution thus obtained (in B), let cool, and acidify with HCl. On now adding a little Fe_2Cl_6 a blood-red colouration is produced, which is destroyed on the addition of a little $HgCl_2$ (solution).

FIG. 4.



This test is very delicate, and may be more simply applied thus :—

6. Boil the cyanide solution with excess of Am_2S (after boiling for a short time the yellow colour should still remain). If a precipitate form, filter, acidify the filtrate with HCl, evaporate nearly to dryness, and add several drops of Fe_2Cl_6. A blood-red colouration is produced, which is destroyed by $HgCl_2$, but not by boiling, or by the addition of HCl. HCl removes the colour formed by the action of Fe_2Cl_6 on a formate or acetate (see **11 and 13**).

7. Cyanides destroy the dark-red colour of I dissolved in KI.

N.B.—Mercury cyanide ($HgCy_2$) does not answer to Tests (1), (2), (3), (4), or (5), but does to (6). When heated as in (1), this cyanide gives off CN gas, which burns with a beautiful purple flame. If the presence of $HgCy_2$ be suspected, to the solution add excess of H_2S; a precipitate of HgS is, of course, formed if mercury is present. Filter, boil the filtrate until there is no further smell of H_2S, and test for a cyanide according to Tests 1–6.

37. *Tests.*—Ferrocyanic Acid.

1. Heated alone, ferrocyanides react like the simple cyanides (see **36**, 1).

2. On adding dilute H_2SO_4 to a ferrocyanide, a bluish-green colour is obtained, which becomes blue-black when the solution is boiled. Heated with strong H_2SO_4, a white precipitate is formed, and HCN is evolved.

Introduce into a small retort some potassium ferrocyanide, acidify strongly with H_2SO_4 (concentrated) and distil over into a small quantity of H_2O. Divide the solution of HCN, thus obtained, into three parts, and test for the presence of a cyanide by **36** (3), (4), (5).

3. Fe_2Cl_6, added to a ferrocyanide solution, produces a dark-blue precipitate of Prussian blue ($Fe_4K_2Cy_{12}$), which is soluble in $C_2O_4H_2$, forming a blue-black liquid; but is insoluble in mineral

acids. On the addition of KHO, the Prussian blue is decomposed, brown $Fe_2(HO)_6$ being separated out.

4. $FeSO_4$ gives a whitish precipitate, which rapidly becomes blue on exposure to the air.

5. $AgNO_3$ gives a white precipitate of Ag_4FeCy_6, *insoluble in AmHO* and in HNO_3, but soluble in KCN. Heat the precipitate of Ag_4FeCy_6 with HNO_3; orange brown $Ag_6Fe_2Cy_{12}$ is formed, which is *soluble in AmHO*. When heated alone, Ag_4FeCy_6 decomposes, metallic Ag being reduced.

6. $CuSO_4$, added to a ferrocyanide solution, gives a chocolate-brown precipitate of Cu_2FeCy_6, which does not dissolve on the addition of $H\bar{A}$.

38. *Tests.*—Ferricyanic Acid.

1. When heated alone, these salts react in the same manner as the simple cyanides (see **36**, 1).

2. Heated with dilute H_2SO_4, a little HCN is given off, and the colour changes from greenish-yellow to very dark brown. When strong H_2SO_4 is used, HCN is readily evolved, as in **37**, 2.

3. Fe_2Cl_6 gives a reddish brown (with a green tint) colouration, but no precipitate.

4. $FeSO_4$ gives a dark-blue precipitate of Fe_5Cy_{12} ('Turnbull's Blue'), insoluble in most acids. On adding KHO, the blue colour is destroyed.

5. $AgNO_3$ gives an orange-coloured precipitate of Ag_3FeCy_6, soluble in AmHO, insoluble in HNO_3.

N.B.—Ferricyanides are very liable to contain

traces of a ferrocyanide, which, of course, give the ferrocyanide reactions in a slight degree.

39. *Tests.*—Sulphocyanic Acid.

1. When heated in a hard tube, sulphocyanides are decomposed, sulphur being separated, but no charring takes place.

2. Heated with strong H_2SO_4, the solution acquires a pinkish tint; small quantities of SO_2 and of HCN are evolved.

3. Fe_2Cl_6 gives a blood-red colouration with sulphocyanides. No precipitate is thrown down, as the sulphocyanide of iron formed is soluble. The colour is destroyed by $HgCl_2$, but not by HCl. The colour obtained by the action of Fe_2Cl_6 on a formate or acetate is destroyed by HCl (see **11**, 4).

4. With $AgNO_3$ these salts give a white curdy precipitate of AgCNS, soluble in AmHO and in KCNS (solution), but insoluble in dilute HCl.

5. $CuSO_4$ produces a black crystalline precipitate of cupric sulphocyanide ($Cu(CN)_2S_2$), if the solution is strong. On standing the precipitate becomes white, the cupric becoming cuprous sulphocyanide ($Cu_2Cy_2S_2$).

40. Alcohol or Ethyl Hydrate: C_2H_6O.

Alcohol (ethyl), the second on the list of the normal primary alcohols, is a colourless, transparent liquid, with a spirituous smell and burning taste. It combines very eagerly with H_2O, evolving heat and undergoing contraction of volume (about 3 per cent.)

When pure it boils at 78·4°; it has not yet been solidified, though it becomes viscid at −105° C.[1] Alcohol possesses a specific gravity of 0·807 at 0° C., and of 0·7938 at 60° F. It burns with a non-luminous blue flame, forming CO_2 and H_2O by its combustion. Alcohol acts as a solvent for many salts as well as gases, and was used for the precipitation of the malates (see **20**, 3); it also dissolves many resins, oils, and bases which are insoluble in water and other common solvents. When acted upon by oxidising agents (HNO_3; $H_2SO_4 + MnO_2$; $K_2Cr_2O_7 + H_2SO_4$, &c.), alcohol becomes converted, first, into aldehyd, $\left.\begin{matrix}CH_3\\COH\end{matrix}\right\}$, and then into acetic acid, $\left.\begin{matrix}CH_3\\CO_2H\end{matrix}\right\}$ (each of which has a characteristic odour), while its vapour, when passed through a red-hot tube, is decomposed with the formation of CH_4, C_2H_4, H, $C_{10}H_8$, C_6H_6, and other compound bodies. When treated with H_2SO_4, hydrogen ethyl sulphate (sulphovinic acid), $\left.\begin{matrix}C_2H_5\\K\end{matrix}\right\}SO_4$, and ether, $\left.\begin{matrix}C_2H_5\\C_2H_5\end{matrix}\right\}O$, are formed.

41. *Tests.*—Alcohol.

1. Introduce into a small retort some dilute C_2H_6O, distil over one-half. To the distillate add excess of solid K_2CO_3; this combines eagerly with the H_2O, and two layers are formed, the upper being alcohol and a

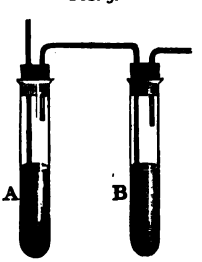
[1] MM. Olzevski and Wroblewski are said to have lately succeeded in solidifying alcohol and carbon bisulphide.

very little H_2O, the lower being a strong solution of K_2CO_3. In order to obtain absolute alcohol (perfectly free from H_2O), the alcohol is distilled in a large retort containing pieces of quicklime.

2. Put two small crystals of I in a test-tube, then enough KHO to dissolve all the I. Add the dark-brown solution thus obtained to a little C_2H_6O, and warm gently. The brown colour disappears, and a yellow crystalline precipitate of iodoform (CHI_3) is obtained, while a sweet smell is evolved. [This test is very delicate, but a large number of other bodies give the same reaction.]

3. In the test-tube A (fig. 5) put some C_2H_6O, and in B two or three drops of $K_2Cr_2O_7$ solution and a quantity of H_2SO_4. Now heat A; the yellow colour of the solution in B becomes grass-green, and a precipitate may be formed. Chromic oxide is formed in the tube B, and dissolves, to a green solution, in the H_2SO_4 present. [This reaction also is given by other combustible vapours.]

FIG. 5.



42. Alcohol, when given for examination purposes, is generally diluted with a considerable quantity of H_2O, so that the liquid is non-inflammable. If, therefore, a solution be given which leaves no residue on evaporation, and if, when distilled as in (1), the

distillate gives the reaction (2) or (3), it may be concluded that alcohol is present.

43. Glycerine: $C_3H_8O_3$ or $C_3H_5\begin{cases}HO\\HO.\\HO\end{cases}$

This substance is a colourless, viscid liquid, possessing a very sweet taste; soluble in alcohol, and in all proportions in H_2O, but insoluble in ether. Glycerine is heavier than H_2O, its specific gravity being 1·27. It can only be distilled in presence of aqueous vapour or in a vacuum, as when heated in the air it undergoes decomposition. When a mixture of $C_3H_8O_3$ and H_2O is heated, the water comes off first. As seen from the above formula, glycerine contains three atoms of hydroxyl, and is therefore capable of holding Cu salts in solution (see **17**).

44. *Tests.*—Glycerine.

1. Heat in an evaporating dish a mixture of glycerine and water; nearly all the water comes off at 100° C., and a viscid liquid is obtained which boils, without evaporating, considerably above 100° C. (proved by dipping a thin test-tube, containing a little H_2O, into the viscid liquid, and showing that the water boils). The liquid is inflammable, burning with a luminous flame, and no residue is left.

2. Heat a little $C_3H_8O_3$ with strong H_2SO_4, or, better, with excess of potassium bisulphate (as SO_2 and other gases are given off from the H_2SO_4, and might disguise the reaction). A stifling smell of acroleine

is perceived (which attacks the mucous membrane of the nose and eyes) while the solution blackens.

$$C_3H_8O_3 - 2H_2O = C_3H_4O \text{ (acroleine).}$$

Any compound of glycerine (fat, tallow, &c.) will react in the same manner.

3. To a little $CuSO_4$ add some KHO; a blue precipitate of $Cu(HO)_2$ is, of course, formed. On now adding glycerine, the precipitate dissolves, and a dark blue solution is obtained.

4. Glycerine, added to a little $K_2Cr_2O_7$ solution and much H_2SO_4, turns the yellow colour of the solution to green on boiling.

Reactions (3) and (4), as before stated, are not characteristic of $C_3H_8O_3$ alone.

The best test for glycerine, so far as the first M.B. student is concerned, is that it is a liquid which boils considerably above 100° C., which leaves no residue and which answers to reaction (2).

45. Chloroform : $CHCl_3$.

Chloroform is a colourless, mobile liquid, possessing a powerful but pleasant smell and a sweetish taste. Its specific gravity at 0° is 1·525, and it boils at 62° C. Chloroform mixes with C_2H_6O in all proportions, but is almost insoluble in H_2O. It is inflammable, burning with a feeble greenish-coloured flame.

46. *Tests.*

1. When a mixture of $CHCl_3$ and H_2O is distilled

in a small retort, the $CHCl_3$ comes off first, and may thus be partially separated from the H_2O.

2. $AgNO_3$ gives no precipitate with a little $CHCl_3$. Heat some $CHCl_3$ with alcoholic potash (solid KHO dissolved in C_2H_6O) and nitric acid ; on now adding $AgNO_3$ a white precipitate is formed.

3. Pass the vapour obtained by heating a mixture of $CHCl_3$ and H_2O through a horizontal tube heated to redness. The $CHCl_3$ is decomposed, and some of the Cl is converted into HCl by combining with the H of the water vapour. The HCl thus formed is led into a little water, and the solution is then tested for a chloride.

Or better :—

4. Act on $CHCl_3$ with some alcoholic potash. On heating, the $CHCl_3$ is decomposed, and KCl is formed in the excess of KHO present. Neutralise the solution with HNO_3, and test the solution for a chloride.

47. Cane Sugar (Sucrose): $C_{12}H_{22}O_{11}$.
Milk Sugar (Lactose): $C_{12}H_{22}O_{11}$.
Grape Sugar (Dextrose): $C_6H_{12}O_6$.
Starch, Cellulose, Dextrin } **(Amyloses)** : $n(C_6H_{10}O_5)$.

These substances belong to a class of bodies commonly called carbo-hydrates, since their formula consists of C united with H and O (in the proportion necessary to form water). They possess the power, in common with many other organic bodies, of deviating

the plane of polarised light. The substances in the above group turn it to the right, and are consequently said to be dextrogyrate. Cane sugar occurs in many plants, as the sugar cane, the sugar maple and beet-root. It also occurs, with grape and fruit sugar, in honey and several kinds of fruit. Cane sugar possesses a sweet taste, which is best observed in solution. It is freely soluble in hot and cold water, but nearly insoluble in C_2H_6O.

Milk sugar (lactose), as its name implies, occurs in the milk of mammals, from which it is obtained by acidifying slightly, evaporating, and allowing to crystallise. It is soluble in H_2O (but not so readily as cane sugar), slightly soluble in C_2H_6O, insoluble in ether. It is not so sweet as cane sugar, feels gritty when tasted, and requires a certain time to show its sweetness.

Grape sugar (dextrose) occurs in ripe fruit, manna, honey, and in diabetic urine. It is not so sweet as cane sugar, but sweeter than lactose, and it does not feel gritty to the taste. It is readily soluble in H_2O, and also to a certain extent in C_2H_6O (more so than cane sugar).

Starch is an indispensable constituent of certain parts of plants, and occurs very widely throughout the vegetable world. It is a white powder, consisting of fine granules, each possessing a distinct organised structure. The size and shape of the granules vary according to the source from which the starch has been obtained. Starch corresponds with cellulose

in composition, but in the vegetable cellulose seems to play merely a mechanical part, undergoing little chemical change, while the chemical changes which the starch undergoes seem to resemble in some measure the process of digestion in an animal. Starch is insoluble in cold H_2O, in C_2H_6O, and ether, but when heated above 60° C. the granules swell and burst, and a substance is obtained which is soluble in cold H_2O, but not in C_2H_6O. Heated to 160° C., starch is converted into dextrin.

Cellulose occurs nearly pure in cotton, linen, and the woody fibre of young plants. It is a white substance resembling starch in composition, insoluble in H_2O, C_2H_6O, or ether, but soluble in a solution of cupric oxide in $\overline{Am}HO$. Cellulose does not swell up or become soluble when immersed in hot H_2O, and is not soluble in KHO, except when the solution (of KHO) is excessively strong, when chemical action takes place.

Dextrin (or British gum) is formed when starch is heated to 160° C. It is readily soluble in H_2O, but insoluble in C_2H_6O. Its specific rotatory power is considerable, being 138·7° to the right. As generally prepared dextrin always contains dextrose, and consequently gives the dextrose reactions.

48. *Table of Differences of the Sugars.*

	Spec. Gr.	Specific Power of Rotation	Structure
Sucrose .	1·606	73·8° +	Rhomboidal prisms, four and six-sided
Lactose .	1·543	59·3° +	Small cubes and square plates
Dextrose	—	56° +	Crystallises with difficulty in cubes

49. *Tests.*—Cane Sugar.

1. Heated alone the solid melts, water is given off, much charring then takes place, and a strong smell of burnt sugar is perceived.

2. Heated with strong H_2SO_4, the mixture chars rapidly, much CO_2, SO_2, and little CO being evolved.

3. To an excess of KHO add a few drops of $CuSO_4$; a blue precipitate is, of course, formed. On now adding solution of cane sugar, the precipitate dissolves and a blue solution is obtained. Boil some of the blue solution thus obtained; either (1) no reduction of the Cu salt occurs, or (2) more commonly there is a slight reduction, because the cane sugar contains traces of grape sugar which reduce the Cu solution, red oxide of copper (Cu_2O) being formed (see **51**, 3).

4. To another portion of the blue solution (in 3), add HCl till the solution is acid, then boil for one or two minutes; the cane sugar is converted into grape

sugar, which reduces the Cu solution with the formation of the red oxide of copper (Cu_2O).

50. Lactose and Dextrose.

These two bodies resemble each other very closely, and it is difficult to distinguish between them. When heated gently with HNO_3, from lactose we get mucic acid ($C_6H_{10}O_8$), which is *slightly* soluble in H_2O, while from dextrose we get saccharic acid ($C_6H_{10}O_8$), which is *very* soluble in H_2O. In the solid form lactose is very gritty and almost insipid, but quite sweet in solution. Dextrose is not gritty, and is much sweeter and more soluble than lactose.

51. *Tests for both.*

1. When heated alone the solid melts, water of crystallisation is given off, much charring occurs, and an unpleasant smell of burnt sugar is perceived.

2. Heated with strong H_2SO_4, combination takes place, a yellow-coloured compound (sulphosaccharic acid) is formed, and no charring occurs.

3. When heated to boiling with a few drops of $CuSO_4$ and excess of KHO, a yellowish red precipitate of Cu_2O is at once produced, due to the reducing action of the grape sugar. This reaction enables us to distinguish between cane and grape sugars.

52. *Tests.*—Starch.

1. When heated alone, much charring takes place, and a pungent smell resembling burnt sugar is given off.

2. Heated with strong H_2SO_4, starch gives the same reactions as cane sugar.

3. Starch, and a solution of starch, give an intensely dark-blue colour (black when much starch is used) with a weak solution of I in KI. The colour is bleached by alkalis and disappears on heating the solution, but reappears, to a certain extent, on allowing the solution to cool.

4. Starch granules are soluble in KHO, forming a gelatinous mass.

5. Starch solution, when boiled with a few drops of HCl, is gradually converted, first, into dextrin (see **54**), and then into grape sugar (dextrose : see **51**).

[When heated with H_2SO_4 dextrin is converted into grape sugar. $C_6H_{10}O_5 + H_2O = C_6H_{12}O_6$.]

53. *Tests.*—Cellulose.

1. Gives no blue colour with a solution of I in KI.

2. A solution of CuO in AmHO dissolves cellulose, but not starch. To the solution thus obtained add HCl; a gelatinous precipitate of cellulose forms in the upper part of the solution.

3. Cellulose is insoluble in KHO solution.

54. *Tests.*—Dextrin.

1. Dextrin gives no blue colour with a solution of I in KI.

2. To a few drops of $CuSO_4$ add an excess of KHO. The blue precipitate thus obtained dis-

solves on adding dextrin solution. Now boil. There should be no reduction, as pure dextrin does not reduce the Cu salt, but generally there is a reddish precipitate, due to the presence of dextrose in the samples of dextrin. Divide the solution (containing the precipitate) into two parts, (α) and (β). To (α) add HCl (to convert the dextrin into dextrose), the red precipitate readily dissolves; boil for a few minutes, make alkaline with KHO, and compare (α) with (β). There should be more precipitate in (α) than in (β).

3. Dextrin is thrown down from its solutions as a white, milky precipitate on the addition of C_2H_6O. When the solution containing the precipitate is evaporated down, a gummy mass is obtained.

55. Albumen: $C_{72}H_{112}N_{18}SO_{22}$ (Johnson).
Gelatin: $C_{41}H_{67}N_{13}O_{16}$.

In all probability these two substances have never been prepared in a pure state, and therefore the above formulæ are probably presumptive. Gerhardt regarded albumen as a compound of sodium having the formula ($NaC_{72}H_{111}N_{18}SO_{22}H_2O$). Albumen occurs in the solids and fluids of mammalia, and may be obtained in a comparatively pure state from the white of eggs. Beat up the white of a few eggs, dilute with a little water, and strain the liquid thus obtained to free it from extraneous matter, as pieces of membrane, &c.

When skin, membrane, or bone is subjected to the

action of H_2O at a high temperature, a colourless liquid is obtained which sets to a jelly on cooling. The substance thus obtained is gelatin. It contains no S, whereas albumen does.

56. *Tests.*—Albumen.

1. On heating a solution of albumen to about 75° C. coagulation at once takes place, and the albumen becomes insoluble.

2. Dilute HNO_3 and most mineral acids cause the coagulation of albumen in the cold. Phosphoric (ortho-), acetic, and tartaric acids do not give this reaction.

3. Add one drop of $Pb\bar{A}_2$ to a solution of albumen; a white precipitate is obtained. Now add excess of KHO; a clear solution is the result (the S in the albumen does not now act on the Pb salt). On boiling, a black precipitate of PbS is formed, owing to the decomposition of the KHO, and the reduction of the lead salt to lead sulphide.

4. Add some albumen solution to a little Millon's reagent;[1] a white precipitate is formed, which becomes red on being heated.

$HgCl_2$ coagulates solutions of albumen, even when very dilute. Other salts (*e.g.* $AgNO_3$, $CuSO_4$, alum, $SnCl_2$) react in the same manner, forming insoluble

[1] Mercury dissolved in HNO_3; the solution thus contains mercurous and mercuric salts, and generally nitrous and nitric acid as well.

compounds with the albumen. Alkalis prevent the precipitation of albumen.

57. *Tests.*—Gelatin.

1. Gelatin does not coagulate on boiling.

2. Does not coagulate on the addition of dilute HNO_3.

3. Gelatin contains no S; consequently there is no formation of a black precipitate of PbS (as in **56**, 3) on adding PbA_2 and boiling with KHO.

4. Gelatin gives a red colour with Millon's reagent.

5. Solution of tannin precipitates both albumen and gelatin. Both hold Cu salts in solution in the presence of an alkali.

A solution of gelatin, when kept, undergoes a chemical change, being gradually transformed into a solid body.

58. *Alkaloids.*

		Source
Morphine:	$C_{17}H_{19}NO_3$, H_2O	Opium, as meconate of morphia.
Quinine:	$C_{20}H_{24}N_2O_2$, $3H_2O$	Cinchona bark.
Cinchonine:	$C_{20}H_{24}N_2O$	Cinchona bark.
Strychnine:	$C_{21}H_{22}N_2O_2$	In the beans of strychnos ignatii, in the upas tree, and in the bark and fruit of strychnos nux vomica.
Brucine:	$C_{23}H_{26}N_2O_4$, $4H_2O$	In false angustura bark, and in nux vomica.

The above substances form part of a series of bodies containing N and possessing high molecular weights. Their constitution has not yet been deter-

mined, neither have they been artificially prepared (with the exception of conine), but they are generally supposed to be substitution compounds of ammonia. They all contain N and some O, and they act as bases, combining with acids to form salts. They possess an alkaline reaction, form double crystallisable salts with $PtCl_4$, and are practically insoluble in H_2O, but readily soluble in hot C_2H_6O. Some of the alkaloids are soluble in ether : *e.g.* quinine, codein, narcotine ; while others are insoluble : *e.g.* morphine, cinchonine, strychnine. These natural bases turn the ray of polarised light, some to the right, others to the left ; they all have a powerful influence upon the animal system. Though the alkaloids themselves are soluble only in a large volume of H_2O, yet their salts are soluble in that solvent.

59. General Reactions.

We must first determine whether or not we have an alkaloid present in the given solution. The presence of an alkaloid may have been indicated in the preliminary examination (**7**, *a*, 3) ; in addition we have two general tests :—

1. A solution of sodic phospho-molybdate gives a white amorphous precipitate with an alkaloid or a solution of its salts.

2. A saturated solution of HgI_2 in KI gives a yellowish-white precipitate with an alkaloid or its salts.

Then, having ascertained that an alkaloid is pre-

sent, to the original solution add AmHO or KHO to precipitate the alkaloid. Dissolve the precipitate in C_2H_6O, add K_2CO_3 to remove H_2O, and pour off the supernatant alcohol solution. We thus get a concentrated solution of the alkaloid itself; evaporate to dryness, and to the solid thus obtained apply the following special tests :—

60. Morphia.

Morphia is very slightly soluble in cold H_2O (1 in 1,000); easily soluble in hot C_2H_6O; insoluble in ether. The hydrochlorate salt ($C_{17}H_{19}NO_3$, HCl, $3H_2O$) is the one which crystallises best.

Tests.—1. Moisten an evaporating dish with a little neutral Fe_2Cl_6. On now adding either solid morphia or its neutral solution, a dark blue colouration is obtained. The colour is destroyed by excess of acid.[1]

2. Strong HNO_3 gives, with solutions containing morphia or its salts, an orange-red colouration. If the solution is dilute, the colour appears only after a time, or on boiling. To the cold solution add $SnCl_2$ or $Na_2S_2O_3$; the colour disappears.

Tests (1) and (2) may also be applied thus :—

To the original solution of morphia or its salt, add Na_2CO_3; shake the solution well; a white crystalline precipitate of morphia is obtained. Filter, and wash

[1] The colour tests for morphia and the other alkaloids are best performed in a porcelain evaporating dish, or on the cover of a porcelain crucible.

the precipitate with a very little H_2O. On moistening the precipitate with a few drops of neutral Fe_2Cl_6, the blue colour is obtained, and the orange-yellow colouration when strong HNO_3 is used.

3. To a strong solution of morphia or of a morphia salt add a very little HIO_3 solution; I is set free and colours the liquid brown. Divide the solution into two parts, (α) and (β). To (α) add a little fresh starch solution; a blue colour, due to the formation of the so-called iodide of starch, is obtained. To (β) add CS_2 or $CHCl_3$; the solution becomes violet-coloured. [Other organic bodies answer to this reaction.]

4. To morphia or one of its salts add a little strong H_2SO_4; a colourless solution is obtained. On now adding HNO_3, or a crystal of $K_2Cr_2O_7$, a beautiful green is produced.

5. A solution of morphia, made alkaline with AmHO, gives a grass-green colouration on being warmed with ammonio-oxide of copper.

61. Quinine.

Solutions of quinine and of its salts are highly fluorescent in the presence of violet rays. Cinchonine solutions are not. Quinine is slightly soluble in H_2O (1 in 900), readily soluble in C_2H_6O and in ether. Concentrated H_2SO_4 and HNO_3 dissolve quinine; with the former a faint yellow-coloured solution is formed, with the latter a colourless solution which becomes yellow on being heated.

62. *Tests.*—1. Add excess of Cl water[1] to an acid solution of quinine or a quinine salt; no apparent change is perceived. Now add AmHO; an emerald-green colour is obtained (see **64**, 2).

2. AmHO (KHO, or Na_2CO_3) gives a white bulky precipitate of hydrate of quinine with a solution of a quinine salt. The precipitate is only slightly soluble in AmHO, less soluble in KHO, but *easily soluble in ether* (see **64**, 3).

3. To a slightly acid solution of quinine or of its salts add Cl water, then some $K_4FeC_6N_6$ and AmHO solution; a fine deep red colouration is produced, which is very liable to disappear.

63. Cinchonine.

Cinchonine has a great resemblance to quinine, differing from it only by containing one atom less of O. It acts as a powerful base, and is less soluble in C_2H_6O than quinine, but its salts are much more soluble, both in H_2O and in C_2H_6O, than those of quinine.

64. *Tests.*—1. Solutions of cinchonine and of its salts are not fluorescent (see **61**).

2. Cinchonine and its salts give no green colouration on the addition of Cl water and of AmHO, but a yellowish-white precipitate is obtained (see **62**, 1).

[1] Instead of Cl bromine vapour may be used, and is said to be more delicate.

3. AmHO (or KHO) precipitates cinchonine as a white amorphous powder from solutions of its salts. The precipitate is *insoluble* in excess of either reagent or *in ether* (see **62**, 2).

4. If to a strong solution of cinchonine, or one of its salts in HCl, excess of $K_4FeC_6N_6$ be added, a yellowish flocculent precipitate is obtained, which disappears on heating. Bright yellow crystals are deposited from the solution on cooling.

65. Strychnine.

Strychnine is soluble in dilute C_2H_6O and in $CHCl_3$, insoluble in ordinary and acetic ether, very slightly soluble in H_2O (1 in 7,000).

66. *Tests.*—The solid strychnine for these tests may be obtained from its solutions, either as directed at **59** or as follows: Precipitate the strychnine by adding AmHO; this will take some time; do not wait, but add $CHCl_3$, which takes up the alkaloid. On evaporating the chloroform solution to dryness, the strychnine is obtained in a solid form.

1. To a little concentrated H_2SO_4 add a trace of solution of $K_2Cr_2O_7$. If a particle of strychnine be moistened with this solution, a beautiful purple colour is produced, quickly fading into violet and red. In place of $K_2Cr_2O_7$ we may substitute MnO_2 (which gives a violet colouration), PbO_2 (which gives a blue colour changing to violet, red, and yellow), or $K_4FeC_6N_6$ (which also gives a violet colour).

This reaction is very delicate, and is highly characteristic of the presence of strychnine.

2. KCyS gives, with even dilute solutions of strychnine, a white precipitate, seen under the microscope to consist of tufts of needle-shaped crystals.

3. Strychnine is soluble in concentrated H_2SO_4 and HNO_3. The former gives a colourless solution which remains unaltered when heated to 100° C.; the latter also gives a colourless solution which becomes yellow[1] when the temperature is raised.

67. Brucine.

Brucine is more soluble in H_2O and C_2H_6O than strychnine.

Tests. 1. To a little solid brucine add a few drops of HNO_3. The brucine dissolves and a bright red colouration is obtained, which becomes blue on the addition of $SnCl_2$ or $(NH_4)_2S$, and yellow on the application of heat.

This reaction is very delicate, and is highly characteristic of brucine. Hence, also, brucine is a very delicate test for the presence of nitric acid.

[1] An orange colour is obtained if brucine be present as an impurity.

Test 1 in **11, 13, 26, 34**, and tests 1 and 2 in **15, 16, 18–21, 25, 30, 31, 36–39, 49, 51, 52** are to be performed on portions of the solid substances.

SECTION III.

GENERAL EXAMINATION OF A SOLUTION FOR AN OXALATE, ACETATE, BENZOATE, SUCCINATE, TARTRATE, CITRATE, MALATE, MECONATE, URATE, HIPPURATE, GALLATE, TANNATE, CYANIDE, OR DOUBLE CYANIDE, OR FOR ALCOHOL, STARCH, CANE SUGAR, GRAPE SUGAR, GLYCERINE, UREA, MORPHIA, QUININE, CINCHONINE, OR STRYCHNINE.

68. This examination should always be preceded by a preliminary one, as directed in **1–9.**

If the solution has left no residue on evaporation, test separate portions of it, at once, for alcohol by **41**, 2 and 3 ; for glycerine by **44**, 2 ; for hydrocyanic acid by **36**, 6 ; or for acetic acid by **13**, 3. If no result be obtained, probably water alone is present.

If a tarry smell, accompanied by much charring, be observed when the solid substance, obtained by evaporation of the original solution, is heated as directed at **7** A, test the original solution at once for an alkaloid.

If a smell of burnt sugar be observed on heating the original solid, test separate portions of the solution at once for cane-sugar by **49**, 3 and 4 ; for grape-sugar by **51**, 3 ; or for starch by **52**, 3.

If no such indication is given, pass on to **69.**

Examination for the Radicles included in Group I.

Oxalates, Meconates, Tartrates, Citrates, Malates.

69. Add to a portion of the original 'Na_2CO_3' solution a little $CaCl_2$ solution; shake well and let stand for a few minutes.

A. *No precipitate is produced.*

The salt is not an **oxalate, tartrate,** or **meconate.** Pass on to **70.**

B. *A white precipitate is obtained in the cold.*

The salt may be an **oxalate, tartrate,** or **meconate.**

Wash the precipitate, and add excess of $H\bar{A}$ to the white precipitate.

(α) *The precipitate dissolves.*

A **tartrate** or **meconate** is present. Test in the original 'Na_2CO_3' solution for a **tartrate** by **19,** 3, and for a **meconate** by **23,** 3.

(β) *The precipitate remains undissolved.*

An **oxalate** is present. Confirm by **18,** 3 and 5.

70. Heat the solution, to which $CaCl_2$ has been added without producing any precipitate, to boiling, and add an equal volume of C_2H_6O.

A. *No precipitate is formed.*

No radicle belonging to Group I. is present. Pass on to **71.**

B. *A precipitate is obtained on boiling.*

A **citrate** is present.[1] Confirm by **21**, 5.

C. *No precipitate is produced until* C_2H_6O *has been added.*

A **malate** is present. [See note 1, page 57.] Confirm by **20**, 5.

Examination for the Radicles included in Group II.

Ferrocyanides, Ferricyanides, Sulphocyanides, Benzoates, Succinates, Hippurates, Acetates, Formates, Gallates, Tannates.

71. Add to a portion of the original 'Na_2CO_3' solution a few drops of Fe_2Cl_6; if there is no precipitate in the cold, heat to boiling.

A. *No precipitate or colouration is produced.*

None of the radicles included in Group II. are present. Pass on to **72.**

B. *A dark-blue precipitate is formed.*

A **ferrocyanide** is present. Confirm by **37**, 3 and 5.

C. *A buff-coloured precipitate is obtained.*

A **benzoate** or **hippurate** is present. Add to a portion of the original solution (concentrated if necessary), or to the buff precipitate, some HCl and shake well. A white precipitate is formed. If, on adding a little ether, the precipitate dissolves, a **benzoate** is present; if the precipitate remains undissolved, a

[1] A malate may be precipitated here if the solution is very concentrated. The reaction with lime water (**20**, **21**, 4) will distinguish between a citrate and malate.

hippurate] is present. Confirm by **15**, 4, and **26**, 2, respectively.

D. *A brownish red precipitate is formed, which is soluble in HCl.*

A **succinate** is present. Confirm by **16**, 4 or 6.

No precipitate is formed in the cold, but

E. *A deep red colouration is produced. No precipitate is thrown down on heating.*

A **sulphocyanide** is present. The colour should be destroyed by the addition of $HgCl_2$, but not by HCl.

F. *A dark-greenish or brown colouration is produced, but there is no precipitate on heating.*

A **ferricyanide** is present. Confirm by **38**, 4.

G. *A deep red colour is observed. On boiling, a light brown precipitate is formed, and the solution becomes colourless.*

The salt is an **acetate** or **formate**.

To the original 'Na_2CO_3' solution add $HgCl_2$.

(α) *No precipitate is formed.* An **acetate** is present.

(β) *A white precipitate is produced.* A **formate** is present.

H. *A dark bluish-black colouration is produced.*

On adding H_2SO_4, the solution becomes yellow. A **tannate** or **gallate** is present.

To a portion of the original 'Na_2CO_3' solution add a little solution of gelatin.

(α) *No precipitate is formed.*

A **gallate** is present. Confirm by **31**, 7.

(β) *A yellowish-white precipitate is produced.*

A **tannate** is present. Confirm by **30**, 4.

Examination for the Radicles included in Group III.

Cyanides, Cane Sugar, Grape Sugar, Starch, Urates, Urea, Alkaloids.

72. To portions of the original 'Na_2CO_3' solution—

(1) Add a few drops of $FeSO_4$ and Fe_2Cl_6, then KHO till the solution is alkaline, and HCl till it is acid (**36**, 3).

A. *A dark-blue precipitate or bluish-green solution*[1] *is obtained.*

A **cyanide** is present. Confirm by **36**, 6.

(2) Add a little $CuSO_4$, then excess of KHO until a dark-blue solution is obtained; boil the mixture.

A. *No precipitate is formed.*

Grape-sugar is absent. Pass on to (3).

B. *A red or yellowish-red precipitate is formed.*

Grape-sugar is present. Confirm by **51**, 2.

(3) Add a few drops of HCl, drop by drop. Shake well.

A. *A white precipitate is gradually formed.*

A **urate** is probably present. Confirm by dissolving the precipitate, after washing it in a few drops of

[1] If the solution is very dilute.

H_2O, in a very little HNO_3; evaporate carefully to dryness, and moisten the residue with a drop of AmHO. A beautiful purple colour should be obtained if a **urate** is present.

B. *No precipitate is formed.*

Boil the solution, to which HCl has been added without producing a precipitate, for a few minutes, then add $CuSO_4$ and KHO as at (2), and boil again.

A. *A red or yellowish-red precipitate is formed.*

Cane-sugar is present. Confirm by **49**, 1.

(4) Add a little weak solution of I in KI.

A. *A dark-blue colour is produced.*

Starch is present. Confirm by **52**, 2.

(5) Add a few drops of $Hg(NO_3)_2$ solution.

A. *A white precipitate is formed.*

Urea is probably present. Confirm by **34**, 3 or 5.

73. If no result has been obtained, probably the solution contains an **alkaloid**, and should be tested as directed in **59**. Morphia is best detected by **60**, 1 and 2; quinia by **62**, 1; cinchonia by **64**, 3 and 4; and strychnia by **66**, 1.

NOTE 1.—A succinate may be precipitated here; if, therefore, the presence of a malate is not confirmed by **20**, 5, a **succinate** is probably present, and will be detected in Group II.

SECTION IV.

EXAMINATION OF ORGANIC MATTER[1] SUPPOSED TO CONTAIN AN ALKALI; OR MERCURY, ARSENIC, ANTIMONY, SILVER, COPPER, ZINC, OR PHOSPHORUS; NITRIC, SULPHURIC, HYDROCHLORIC, OXALIC, OR HYDROCYANIC ACID; STRYCHNIA, MORPHIA, CHLORAL, ANILINE, PHENOL, OR NITRO-BENZOL.

74. In the presence of organic matter the various reactions generally afforded by the above substances will, in most cases, be either partially or entirely concealed or prevented by the relatively large quantity of organic matter present. Certain operations have therefore to be performed before the substance can be obtained in a proper condition for the application of the usual tests.

Preliminary Examination.

75. Make a careful examination of the physical properties of the mixture, noting down its colour, smell, appearance, &c. The presence of **hydrocyanic acid** is sometimes detected by its smell of bitter almonds,

[1] The organic matter may consist of the coats or contents of a stomach, liver, or other organ or tissue, of vomited matter, food, medicine, beer, wine, spirits, or infusion of tea, coffee, &c.

and the presence of **phosphorus** by its characteristic unpleasant smell. Organic matter containing phosphorus is often luminous when viewed in a dark room. Note the occurrence of any foreign substance, such as particles or crystals of antimony, arsenic, copper, or other salt; also any unusual or suspicious appearance of the organ, tissue, or infusion.

76. If the mixture is liquid or semi-solid, test portions of it with litmus-paper.

A. *The liquid is neutral, or nearly so, to test-paper.* Pass on to **78.**

B. *The liquid reddens blue litmus-paper.*

Probable presence of a **hydrogen** salt. If the liquid is strongly acid, and if much effervescence is caused on the addition of solution of Na_2CO_3, probably a large quantity of **HCl**, $\mathbf{H_2SO_4}$, or $\mathbf{HNO_3}$ is present. Confirm by applying the usual tests for each acid to small portions of the original liquid, disregarding mere traces of their presence.

C. *The liquid turns reddened litmus-paper blue.*

If the liquid requires much acid before neutralisation is effected, a poisonous quantity of **KHO** or **NaHO** is present.[1] The presence of K or Na is proved by the usual tests.

77. Various methods of procedure may sometimes be adopted, and much is left to the ingenuity of the student; thus, if the mixture is only semi-fluid, a

[1] AmHO would have been detected by its smell.

comparatively clear liquid may be obtained by allowing the mixture to remain for some hours in a deep and narrow beaker, or in a test-glass.

General Examination.

78. The examination for **phosphorus** and **hydrocyanic acid** should be proceeded with at once, as these bodies are very liable to undergo chemical change, especially in the presence of organic matter.

79. Phosphorus.

Phosphorus in combination is not a poison,[1] but when uncombined it is. Its presence may be detected thus :—

1. A portion of the organic mixture is placed in a flask, KHO is added and heat is applied. Let the gas which is evolved pass through a glass tube, in which some blotting-paper, moistened with $AgNO_3$, has been inserted. If **phosphorus** is present, a black precipitate of silver phosphide will be formed on the paper. Remove the paper, and put it in a small test-tube with a little Cl water ; the silver phosphide dissolves and a white precipitate of AgCl is formed, while phosphoric acid remains in solution. Filter, and test the filtrate for a phosphate in the usual manner.

[1] The alkaline orthophosphates, however, are said to be non-poisonous, while the similar salts of pyrophosphoric and metaphosphoric acids are said to be poisonous. (Gamgee.)

2. **Introduce** a portion of the organic matter into a flask, containing some H_2SO_4 (dilute) and fitted with a long, wide glass tube. Heat the mixture to boiling. If **phosphorus** is present, the column of steam in the tube is seen to be phosphorescent, when viewed in a dark room. If much phosphorus is present, a lambent greenish-coloured flame may be seen in the tube.

This reaction is very delicate, being given with even a mere trace of P.

80. Hydrocyanic Acid.

Introduce a portion of the mixture into a small retort, the neck of which dips into a little H_2O contained in the receiver. Distil slowly, and test separate portions of the distillate for a **cyanide** by **36**, 4, 5. If no results are obtained, test the distillate for **aniline** by **95**.

81. Oxalic Acid.

To the clear liquid, obtained from the original mixture, add excess of $Pb\bar{A}_2$; a white precipitate of lead oxalate will be obtained if oxalic acid is present. Introduce the precipitate (after washing it with H_2O) into a small beaker or large test-tube containing some H_2O. Pass H_2S through the liquid for a few minutes. A black precipitate of PbS is thus formed, while oxalic acid is set free and enters into solution. Boil the filtrate until it ceases to smell of H_2S, and test the clear liquid for an **oxalate** by **18**, 4.

82. Examination for Hg, Pb, Ag, Cu, As, Sb, Zn.

Test a portion of the mixture with H_2S. If the organic mess is white, the coloured sulphides are readily seen, and thus a slight indication of the metal present is obtained.

Then proceed to destroy the organic matter present. To do this, to a portion of the original mixture add an equal volume of concentrated HCl, and, from time to time, a few crystals of $KClO_3$. Heat to boiling, replacing the water lost by evaporation, until a clear yellow solution,[1] with or without a precipitate at the bottom, is obtained. Add two or three drops of H_2SO_4, and heat the solution to drive off excess of Cl. By this mode of procedure we obtain **AgCl** and **$PbSO_4$** as precipitates. Filter, and test the residue for **Ag** and **Pb**. Pass H_2S through the warm filtrate, and test the precipitate for Hg, As, Cu, Sb, in the usual manner.

83. If any indication of the particular metal present has already been obtained, we may proceed thus :—

For Cu.

Introduce a piece of clean and bright iron (a knife blade will do well) into a little of the 'original HCl solution;' a deposit of Cu forms on the iron if a **Cu**

[1] This solution will be referred to hereafter as the 'original HCl solution.'

salt is present, an equivalent of iron entering into solution at the same time.

Or thus :—Place a piece of Pt foil in an evaporating dish, containing some of the 'original HCl solution' somewhat diluted with H_2O. On the Pt a small piece of zinc is placed. Hydrogen is evolved, and a deposit of Cu forms on the Pt foil, if the organic mess contained a **Cu** salt. The deposit is readily soluble in HNO_3, and the solution gives the ordinary reactions for copper.

If **Sb** is present in the solution, a black deposit will be obtained (on the foil), which is insoluble in cold HCl, readily soluble in warm HNO_3, and which becomes orange-coloured when heated with a drop or two of $\overline{Am}_2S$.

84. For As, Sb, Hg.

Heat a few pieces of clean and bright Cu foil[1] in some of the 'original HCl solution;' the above metals, if present in the solution, will be deposited on the foil. Remove the Cu, and after washing with a little H_2O, carefully dry it on blotting-paper, or at as *low a temperature* as possible (so that the metals may escape volatilisation). Introduce some of the foil into a rather long and narrow sealed tube of hard glass, and apply a gentle heat to the bottom of the tube.

[1] For the foil we may substitute some bright Cu wire made into a small bundle.

A. *A bright white sublimate is formed, readily seen, by the aid of a lens, to consist of liquid metallic globules.*

A **mercury** salt is present in the mixture. The deposit on the Cu, when rubbed, becomes silvery white, and is soon volatilised on the application of heat.

B. *A white or greyish-white sublimate* (As_2O_3) *is formed* (*at some distance from the foil*). *Small sparkling transparent crystals may be seen in the sublimate with the aid of a lens.*

An **arsenic** salt is present in the mixture.

The crystals are octohedral with square bases, and are formed by the oxidation of the As deposited on the foil. The sublimate is soluble in boiling H_2O, and the solution gives a light yellow precipitate with *ammonio-nitrate of silver.*[1]

C. *A white amorphous sublimate is formed near the Cu.*

Probably an **antimony** salt is present in the mixture.

The sublimate of Sb_2O_3 is insoluble in boiling H_2O.

[The As deposit requires less heat for volatilisation than the Sb deposit, and is consequently found at some distance from the source of heat.]

[1] This solution may be obtained by cautiously adding very dilute $\overline{Am}HO$ to a solution of $AgNO_3$, until the precipitate, at first formed, is redissolved.

Confirm the results of A, B, or C by the application of the usual tests for Sb, As, and Hg to portions of the 'original HCl solution.'

85. For As and Sb.

Add KHO to the organic mess, boil down to a thick liquid, pour in HCl until the solution is acid; introduce the mixture into a retort, and distil over. Test the distillate for **arsenious acid**, and the residue for **Sb.**

86. For Zinc.

To the filtrate obtained after the removal of the precipitate produced by the addition of H_2S (**82**), add $\overline{Am}HO$ till alkaline. A white or dark-coloured precipitate is formed. Dissolve the precipitate, after washing, in a few drops of HCl, add a little HNO_3, heat to boiling; now make alkaline again with $\overline{Am}HO$, filter (if a precipitate forms), and to the filtrate add $\overline{Am}_2S$. If **zinc** is present in the solution, a white precipitate will now be obtained.

87. Examination for Strychnia and Morphia.

If the original liquid [1] is acid, add Na_2CO_3 until it is slightly alkaline. Shake up well with $CHCl_3$ in a stoppered bottle for about five minutes, and set by

[1] If the original mixture is solid or semi-solid, heat the mess with H_2O and $\frac{1}{5} - \frac{1}{8}$ its volume of HCl till liquid, then filter, and proceed as above.

till the chloroform has settled. Pipette off (or pour off) the supernatant liquid, and wash the chloroform with H_2O. Pour off the H_2O carefully, and evaporate chloroform solution to dryness on a water-bath. If the solution is not clear when evaporated nearly to dryness, add $CHCl_3$, wash again, and evaporate down. To the residue add a few drops of H_2SO_4 and a trace of $K_2Cr_2O_7$; an *evanescent purple* colour is seen if **strychnine** is present.

To the liquid that was poured off from the $CHCl_3$, add C_2H_6O and solid K_2CO_3; shake well, and set by till the C_2H_6O solution has formed at the top. Carefully remove this layer, introduce it into a small retort, and distil nearly to dryness. Add acetic ether and evaporate down. A *deep blue* colouration, on the addition of neutral Fe_2Cl_6 solution to the residue, indicates the presence of **morphia**.

The separation of morphia from other organic matter is rather difficult; consequently the operation should be conducted carefully, and with as much of the original mixture as can be spared.

88. Chloral (Trichlor-aldehyde) : $\left.\begin{matrix}CCl_3\\COH\end{matrix}\right\}$.

Chloral is a colourless, oily, powerfully smelling liquid, boiling at 98·5°, and possessing a specific gravity of 1·502. When treated with H_2O, heat is evolved and a white crystalline body, chloral hydrate (C_2HCl_3O, H_2O), is formed. When impure, chloral

hydrate is of a dirty yellow colour. Both chloral and its hydrate are soluble in H_2O, C_2H_6O, and $C_4H_{10}O$.

When treated with an alkali, chloroform and the formate of the metal are formed, thus :—

$$C_2HCl_3O + KHO = KCHO_2 + CHCl_3.$$

This reaction is the basis of the following method for detecting the presence of chloral.

89. Distil a portion of the organic matter supposed to contain chloral. To the distillate add KHO, and distil again. If chloral is present, chloroform passes over into the receiver and potassium formate is left in the retort.

Test the distillate for chloroform by **46, 2**, and the residue for the presence of a formate by **11, 4**. If *both* these reactions are obtained, the presence of **chloral** in the original mixture is indicated.

90. Phenol (Carbolic Acid) : $C_6H_5(OH)$.

Phenol is a white, crystalline, deliquescent solid, fusing at 42°, and boiling at 185°. The crystals possess a specific gravity of 1·065 and are soluble in the alkalis, with the production of the phenate of the alkali metal. Phenol is slightly soluble in cold H_2O ; more soluble in boiling H_2O.

91. *Tests.*

1. With phenol neutral Fe_2Cl_6 gives a colouration which is precipitated on adding $H\bar{A}$.

2. Bromine water gives a pale yellow precipitate.

3. With Millon's reagent a reddish colour is obtained.

4. Ammonia (or better, aniline) mixed with phenol solution, and then with a little solution of sodium (or potassium) hypochlorite, gives a blue colouration.

The same colour is obtained by immersing a piece of deal first into phenol solution, then into HCl, and afterwards exposing it to the air.

5. Phenol possesses a peculiar well-known smell and burning taste.

When passed over heated zinc (dust) phenol vapour is converted into benzol, which, when treated with strong HNO_3, gives us nitrobenzol, *q.v.*

92. Nitrobenzol: $C_6H_5(NO_2)$.

Nitrobenzol is a liquid possessing an odour resembling that of the oil of bitter almonds; hence nitrobenzol is sometimes termed the 'artificial oil of almonds.' The compositions of the two bodies are, however, very different: the oil of almonds being hydride of benzol, while nitrobenzol is a substitution product of benzol, in which one atom of hydrogen is replaced by NO_2. The specific gravity of nitrobenzol is 1·20–1·29. In the presence of nascent hydrogen, nitrobenzol is converted into aniline, thus:—

$$C_6H_5(NO_2) + 3H_2 = C_6H_5(NH_2) + 2H_2O.$$

This reaction is the basis of the following process

for the detection of nitrobenzol in the presence of organic matter.

93. Introduce a portion of the original mixture into a retort containing some iron filings and acetic acid. Distil carefully, and let the distillate run back into the retort by holding the neck of the retort perpendicularly. When all the nitrobenzol is supposed to be reduced (after about five minutes), let the distillate pass over into the receiver and test the contents for **aniline**, *q.v.*

94. Aniline (Amido-benzol): $C_6H_5(NH_2)$.

Aniline is a colourless oily liquid of peculiar odour, very slightly soluble in H_2O, readily soluble in C_2H_6O, and $C_4H_{10}O$. Its specific gravity is 1·034, and it boils and may be distilled at 182°. Though neutral to test paper, aniline combines with acids, forming definite salts; thus we have aniline chlorate, $C_6H_5(NH_2)HCl$. With H_2SO_4 aniline forms crystals of aniline sulphate, which, when treated with KHO, are decomposed, aniline being set free.

95. *Tests.*

1. With Fe_2Cl_6 aniline gives a bright purple colouration.

2. With bleaching powder also aniline gives a purple colouration, which is destroyed by excess of the reagent.

3. To a solution of potassium (or sodium) hypochlorite add a trace of aniline; a splendid violet

colour (mauve) is obtained. This reaction is very delicate, and is afforded by a very small quantity of aniline.

4. To a dilute solution of potassium bichromate add a little aniline sulphate ; the colour 'mauve' is produced.

[For fuller information on this subject, a manual of Toxicology should be consulted.]

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